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## MICROSTRUCTURE ANALYSIS OF BORON NANOTUBES

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U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT AND  
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Munitions Engineering Technology Center

Picatinny Arsenal, New Jersey

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## INTRODUCTION

Boron nanotubes (BNT) have never been produced in a reliable manner, yet they remain a promising material worthy of study due to the fact that they are lighter than carbon nanotubes and more galvanically inert. This report will provide transmission electron micrographs (TEM) and corresponding selected area electron diffraction (SAED) patterns obtained from BNT. This study will confirm the success of the production methods used by Universal Global Products (UGP, LLC) to manufacture BNTs consistently. The UGP previously produced B-nanoparticles in hexane solution, which was confirmed by high resolution TEM (ref. 1).

## BORON NANOTUBE THIN FILM PREPARATION TECHNIQUE

The thin film of boron and the nanotubes are produced by the CVD technique and deposited on 600-mesh nickel (Ni) grids. The thin film was also evaporated and deposited on a rectangular glass plate. By using various gas compositions, pressures, temperatures, and substrates/dopants UGP, LLC was able to produce the BNT and thin films in a reliable manner. This work was documented in Small Business Innovation Research (SBIR) Topic A07-031 via monthly reports.

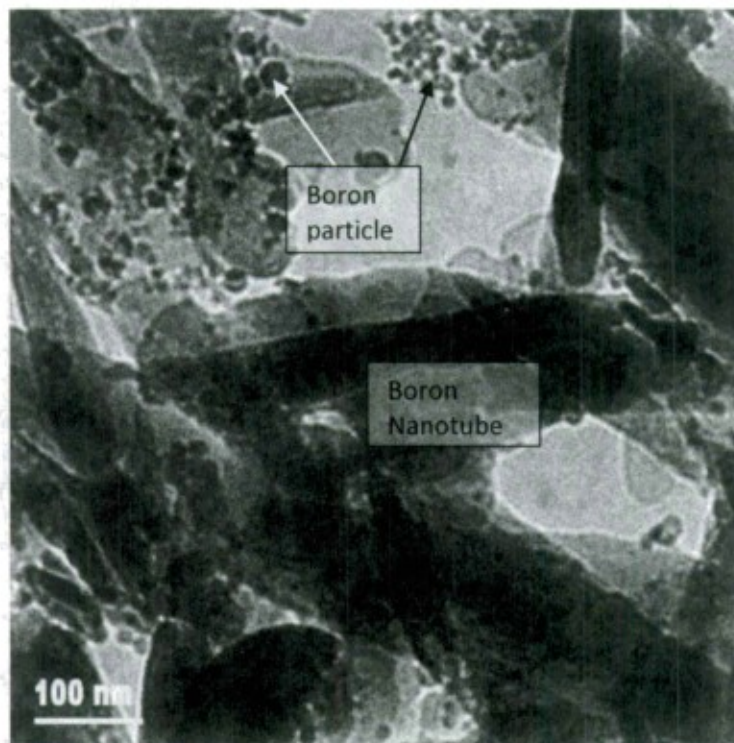
## TRANSMISSION ELECTRON MICROSCOPY SPECIMEN PREPARATION

The UGP, LLC provided the U.S. Army Armament Research, Development and Engineering Center (ARDEC), Picatinny Arsenal, New Jersey a small broken piece of 600-mesh nickel grid with boron thin film on it. This piece was sandwiched between two 400-mesh grids to prevent the sample from falling inside the TEM collimator. The other thin film sample on a glass plate was floated on the surface of distilled water, picked up with tweezers, and placed on a 400-mesh carbon coated copper grid. The Philips 420 electron microscope at 120-KV voltage was used for TEM analyses.

## RESULTS AND DISCUSSION

### Transmission Electron Microscopy Analyses

Figure 1 shows a large number of BNTs. The width of these nanotubes is approximately 25 to 50 nm with different lengths. The white arrow indicates a single boron particle of 5 to 10 nm size. The area indicated by the black arrow reveals a large number of such particles in a group. These particles originated during the evaporation process. Figure 2 reveals a SAED pattern from an area in figure 1. The spotty ring pattern confirms the crystalline structure of the boron particles and nanotubes. This diffraction pattern is difficult to interpret and thus the author cannot confirm the exact phase of this material. Figure 3, taken from a different area, also show similar nanotubes. Elongated micro-BNT filaments are observed at a broad area of the specimen. A SAED resulting from these BNT filaments revealed exactly the same electron diffraction pattern as in figure 3. Figures 5 and 6 are comparatively very low magnification electron micrographs revealing dark nanotubes.



The white arrow indicates a single boron particle. The black arrow shows the area where a large number of boron particles are accumulated. Several nanotubes of different widths and lengths are shown in the picture.

Figure1  
Boron thin film on 600-mesh Ni grid

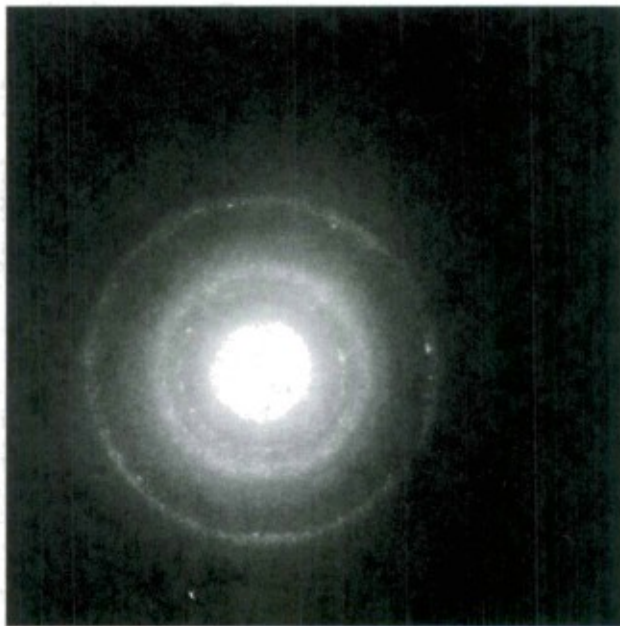
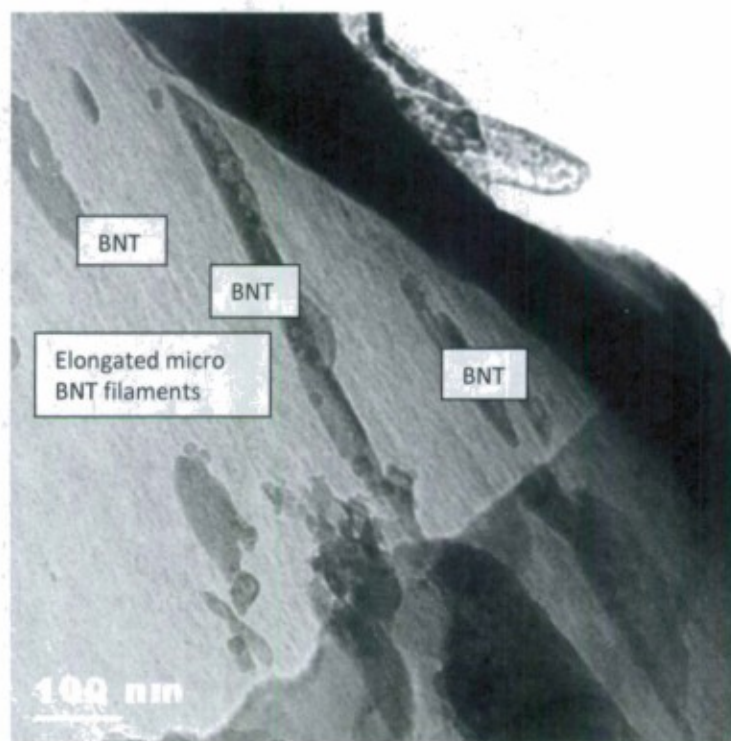


Figure 2  
SAED from an area in figure 1



This picture also reveals the growth of BNTs out of those small filaments.

Figure 3  
High resolution TEM pictures showing very small elongated BNT filaments

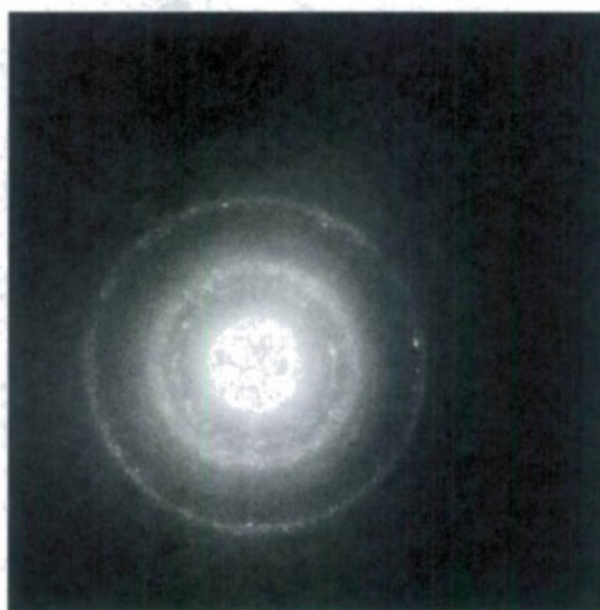


Figure 4  
SAED from an area in figure 3 showing exactly the same kind of spotty rings as in figure 2



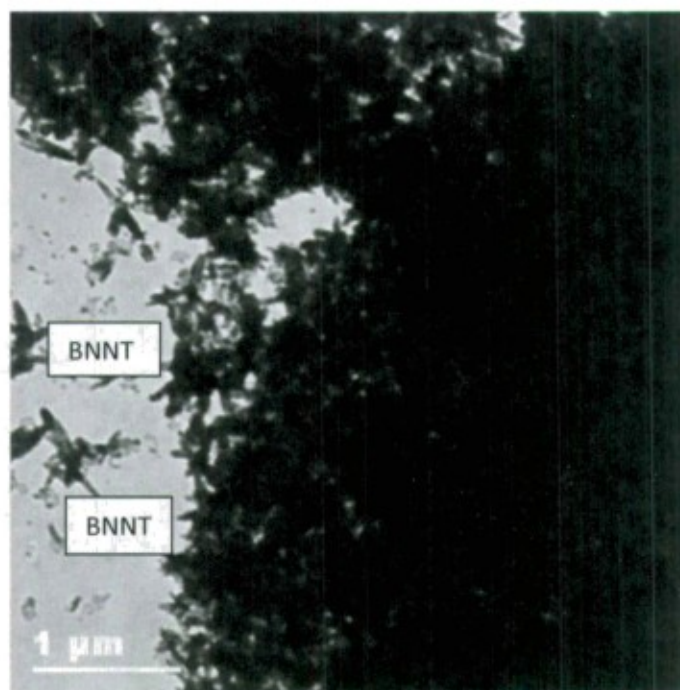


Figure 5  
Low magnification TEM pictures revealing large numbers of dense nanotubes

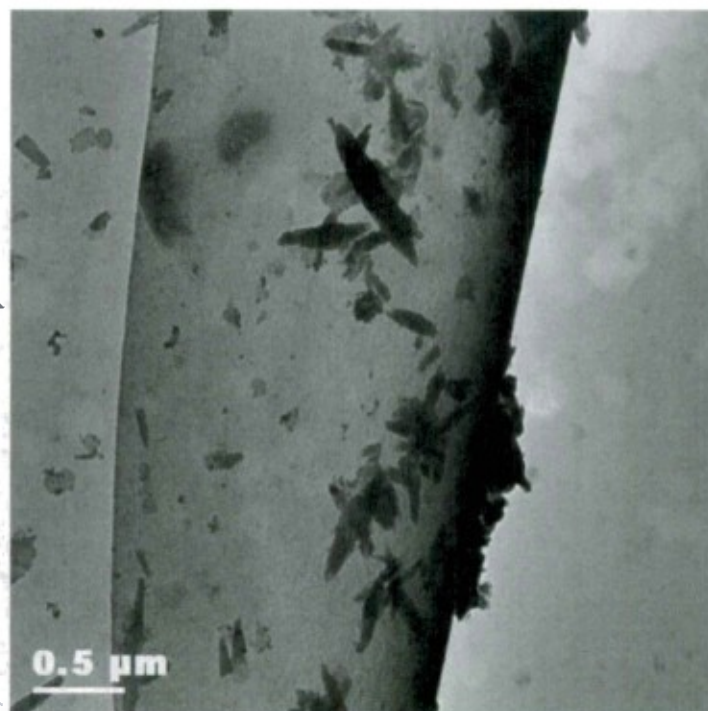
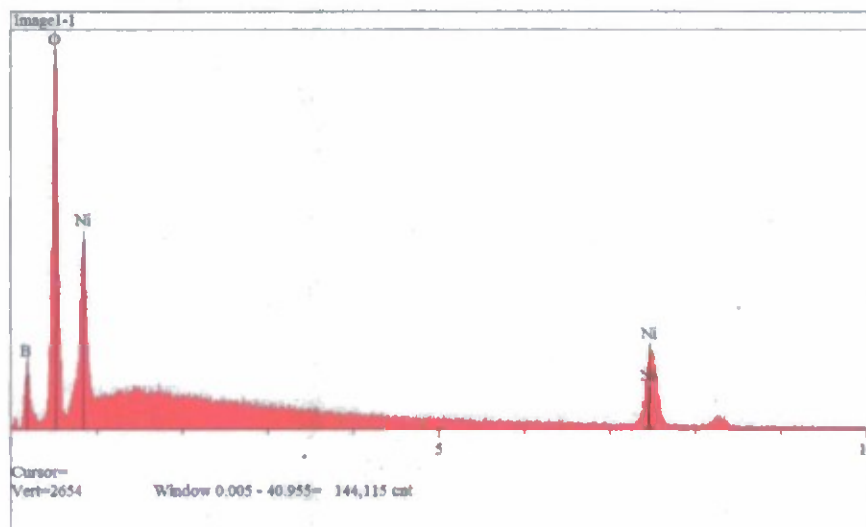


Figure 6  
TEM picture at much lower magnification showing scattered large numbers of BNTs

## Scanning Electron Microscope Analyses

The SEM picture (1000X) taken from the BNT specimen on a folded 600-mesh nickel grid show the BNT thin film supported on the grid (fig. 7). The electron dispersion spectroscopy (EDS) analyses from the area marked with a CROSS show the peak from boron (B), oxygen (O), Ni (K-alpha), and Ni (K-Beta). A low magnification SEM picture (fig. 8) confirms the 600-mesh nickel grid is folded to support the thin boron nitride thin film.



Ni (K-alpha) and Ni (K-beta) originated from Ni grid

Figure 7  
EDAX analyses of boron nitride nano powder showing boron and oxygen

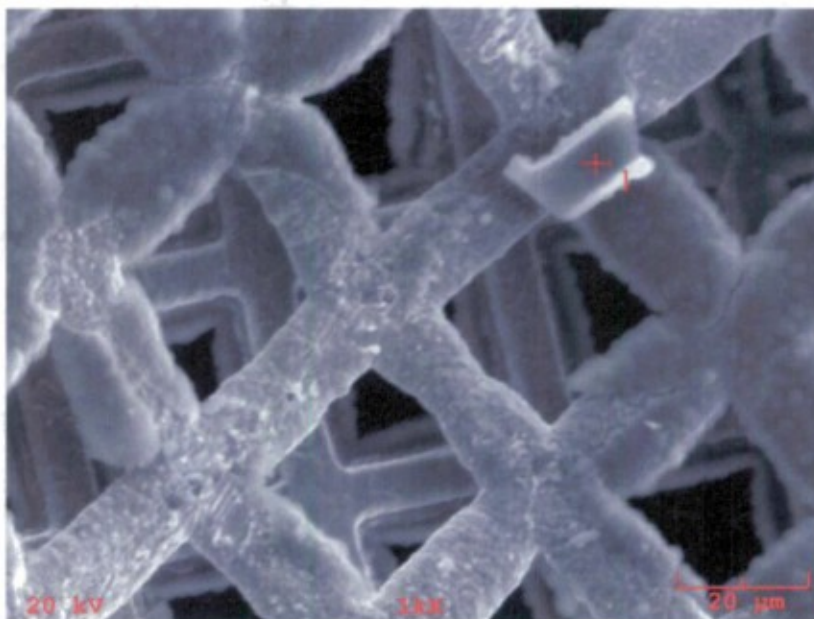


Figure 8  
Low magnification SEM picture of folded 600-mesh Ni grid supporting boron nitride thin film

## CONCLUSIONS

The boron evaporated thin film revealed a large number of nanotubes and nanoparticles. These nanotubes are 25 to 50 nm width with different lengths. The boron nanoparticles have spherical shapes with 5 to 10 nm diameters. The boron nanoparticles filaments could be transformed into nanotubes with proper substrate and evaporation temperature. The x-ray diffraction and Fourier transform infrared analyses will confirm whether this thin film has a boron or mixed-boron molecular structure.

## REFERENCE

1. Chatterjee, Tapan; Kerwien, Stacey; and Jelis, Elis; "Transmission Electron Microscopy of B-Nanoparticles in Hexane Solution," Technical Report ARMET-TR-09038, U.S. Army Armament Research, Development and Engineering Center, Picatinny Arsenal, New Jersey, September 2009.



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